

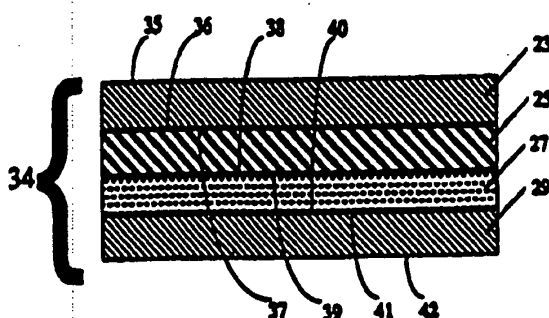
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| <p>(54) Title: MICROPOROUS FILM/NONWOVEN COMPOSITES</p> <p>(57) Abstract</p> <p>A composite comprising four layers adhesively bonded together exhibits a balance of breathability, barrier, strength, comfort, and appearance properties, making the composite ideally suited for a variety of medical, hygiene and sportswear uses. The composite comprises internal webs of cotton (27) and a microporous film (25) flanked by surface webs of nonwoven material (23, 29).</p>   |   |  |

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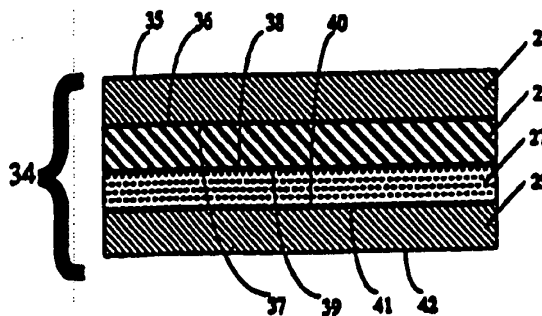
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(54) Title: MICROPOROUS FILM/NONWOVEN COMPOSITES

(57) Abstract

A composite comprising four layers adhesively bonded together exhibits a balance of breathability, barrier, strength, comfort, and appearance properties, making the composite ideally suited for a variety of medical, hygiene and sportswear uses. The composite comprises internal webs of cotton (27) and a microporous film (25) flanked by surface webs of nonwoven material (23, 29).



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## MICROPOROUS FILM/NONWOVEN COMPOSITES

## BACKGROUND OF THE INVENTION

This invention relates generally to  
5 microporous/nonwoven composites which exhibit  
breathability and good barrier properties. In one aspect  
it relates to a composite comprising a nonwoven layer, a  
microporous layer, and a staple fiber layer. In another  
aspect, the invention relates to a method of laminating  
10 webs comprising a nonwoven web, a microporous web, and a  
staple fiber web.

Microporous films (or membranes as they are  
frequently referred to) have long been used in  
applications requiring both breathability (or water vapor  
15 transmissibility) and barrier to liquids. Commercially  
available microporous films include Celgard 2400  
polypropylene film, EXXAIRE polyethylene film produced by  
Exxon Chemical Company, and TetraTex, a microporous  
polytetrafluoroethylene film produced by TetraTek  
20 Corporation, and Gore-Tex produced by W. L. Gore &  
Associates. Other microporous films include those made  
from polyamides, polyesters, polyurethane, and  
polypropylene.

Nonwoven webs, because of their high porosity, are  
25 highly breathable but exhibit little or no barrier  
properties. Efforts have been made to combine nonwovens  
with microporous film to arrive at a composite which  
possesses a balance of breathable and barrier properties.

Such a composite has uses in health care, protective  
30 apparel, footwear, etc.

A paper entitled "EXXAIRE PLUS NON-WOVENS - MADE  
FOR EACH OTHER" was presented at the First Annual TANDEC  
Conference during October 22-25, 1991 in Knoxville,

Tennessee. This paper disclosed a two layer composite comprising a microporous film and a nonwoven HDPE.

An article appearing in Nonwovens Industry dated June 1991, page 38, and entitled "New Light-weight Film  
5 Creating Markets for Nonwoven Composites" discloses a no-heat process for laminating a microporous film to nonwovens using discrete bonding patterns of an adhesive.

Patents which disclose microporous films and microporous film composites include the following:

- 10 (a) U.S. Patent 4,777,073 discloses a breathable polyolefin film prepared by melt embossing a highly filled polyolefin film which is stretched to impart greater permeability to the film;
- 15 (b) U.S. Patent 4,929,303 discloses a breathable poly-olefin film heat laminated to a nonwoven HDPE fabric.

Nonwoven composites are described in the following

U.S. Patents:

- 20 (a) U.S. Patent 4,929,303 discloses a composite comprising breathable polyolefin microporous films and nonwoven fabrics.
- (b) U.S. Patent 4,041,203 discloses a composite of continuous filaments mat thermally bonded to  
25 a web of discontinuous filaments.
- (c) U.S. Patent 4,142,016 discloses a multi-layered structure having a layer of textile fibers and a layer of staple short fibers bound together by a bonding agent.
- 30 (d) U.S. Patent 4,194,939 discloses a composite comprising layers of bulk fibers bonded between two reinforcing textile layers.
- (e) U.S. Patent 4,675,226 discloses an inner layer of cellulose fibers and outer layers of a

continuous filament thermoplastic meltblown fibers or rayon fibers. The layers are stitch-bonded together.

- 5 (f) U.S. Patent 4,950,531 discloses a composite of a meltblown fiber layer and a nonwoven material such as pulp fibers, staple fibers, meltblown fibers, and continuous filaments. The layers are hydraulically entangled together.
- 10 (g) U.S. Patent 4,970,104 discloses at least two non-woven webs bonded together by entangled bonding in spots by jet treatment.
- 15 (h) U.S. Patent 5,149,576 discloses a composite structure comprising nonwoven webs joined together by a mixture of an additive and a thermoplastic polymer.
- 20 (i) U.S. Patent 5,178,931 discloses a composite structure comprising three nonwoven layers of different filament diameters. The boundary between adjacent nonwoven layers is treated with an agent and the three-layered structure is bonded by the application of heat and pressure.
- 25 (j) U.S. Patent 5,200,246 discloses a composite comprising continuous longitudinally extending fibers bonded to webs of nonwovens such as spunbonded webs, meltblown webs, air laid webs, hydroentangled webs, film spun laced webs, etc. The webs are bonded together by meltblowing adhesives at the
- 30 interfaces.
- (k) U.S. Patent 5,230,949 discloses microporous fibers and filaments that can be used alone or

in combination with other materials to form nonwoven web laminates.

(l) U.S. Patent 5,236,771 discloses a composite lining fabric for apparel use comprising a layer of melt-blown fibers bonded to a nonwoven layer of staple fibers or filaments (point bonded and fluid jet entangled).

(m) PCT Application PCT/US93/01783 discloses a multi-layered nonwoven composite comprising a layer of meltblown fibers, a layer of spunbonded fibers, and a layer of staple fibers such as cellulosic based fibers thermally bonded together.

#### 15 SUMMARY OF THE INVENTION

The present invention relates to composite web structures which are breathable and possess good strength and barrier properties. In addition, the composite constructed according to the present invention exhibits desirable aesthetic and comfort properties.

Although the composite of the present invention has a wide range of uses where breathability and barrier properties are necessary, it is particularly adapted for use as protective apparel.

The composite of the present invention comprises, in its broadest embodiment, a three-layered structure having a core layer of staple fiber web, flanked by, and adhesively bonded to, a microporous film, and a nonwoven web.

In a preferred embodiment of the invention, the composite comprises the following four layers:

- (a) a first nonwoven web, preferably a meltblown or spundbond web;

- (b) a microporous film adhesively bonded to the nonwoven web (a);
- (c) a staple fiber web, preferably cotton staple fibers, adhesively bonded to the microporous film; and
- (d) a second nonwoven web, preferably a meltblown web, adhesively bonded to the staple fiber web.

The structure combines the barrier properties of the microporous film, the breathability and strength of the non-woven webs, and the comfort and wicking properties of the staple fibers. The nonwoven webs also improve the aesthetics (appearance and soft hand) and add to the comfort for the user. Tests have also shown that the nonwoven webs, particularly meltblown webs, contribute to the barrier properties of the composite. The staple fiber web which exhibits hydro-philic characteristics, provides a wicking or reservoir layer for moisture and aqueous liquids.

The preferred method of laminating the webs to form the composite involves the steps of (a) applying a thin coat of an adhesive onto the webs so that the adhesive is at the interface of each web to form a composite and (b) feeding the composite into the nip of calender rolls maintained at a low temperature (e.g. less than 100°C) to pressure bond the webs together. The method may be carried out in one pass through the calender wherein all four layers are pressure bonded together, or in two or more passes through the calender wherein two or three layers are bonded together in one pass followed by the addition of one or more layers in subsequent passes.

A particularly surprising aspect of the present invention is that it produces composite structures that are capable of passing both the Blood and Viral



Penetration Tests for protective clothing materials under ASTM Designations ES 21-92 and ES 22-92, respectively.

## 5 BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a schematic illustrating the lamination process for making the composite of the present invention. Figure 2 is an enlarged cross-section of a composite of the present invention.

10

## DESCRIPTION OF THE PREFERRED EMBODIMENTS

The composites of the present invention exhibit both breathability and resistance to liquids, and are useful in a variety of medical and/or hygiene applications such as protective apparel, wound dressings, sterile dressings, absorbents (e.g. diapers), face masks and the like. Composites used in many of these applications should have the following properties:

- (a) breathable,
- 20 (b) resistance to liquid,
- (c) relatively high strength,
- (d) wear resistant,
- (e) comfort,
- (f) appearance, and
- 25 (g) relatively low cost.

In some of the applications, the composite should in addition have absorbency for liquids.

The composite constructed according to the present invention combines the barrier properties of microporous film, the breathability, appearance and strength of nonwovens, and the absorbency and comfort of cotton.

In its broadest embodiment, the composite comprises a core web of staple fibers which exhibits hydrophilic wetting characteristics, and flanking layers of a

microporous film and a nonwoven web. Each of the layers of the three-layered structure are adhesively bonded together by a low temperature, low pressure bonding process.

- 5 In a preferred embodiment, the composite further includes a second web of a nonwoven adhesively bonded to the outer layer of the microporous film. The description of each of the webs used in the composite, the method of lamination, and properties of the composite, are  
10 described below.

#### Nonwoven Webs

- Nonwoven webs are webs made of randomly oriented fibers or filaments of thermoplastic polymer by entangling the fibers or filaments through mechanical,  
15 thermal, or chemical means. Nonwovens exclude paper and products which are woven, knitted, tufted, or felted by wet milling. The preferred nonwoven webs for use in the present invention are the spunbonded and melt-blown webs.

- The spunbonded webs are formed by filaments that  
20 have been extruded, drawn, laid on a continuous belt, and then immediately thermally bonded by passing through a heated calender. These webs are continuous filament fiber structures having an average fiber diameter between 12 and 50 microns.

- 25 Meltblown webs are made by extruding a molten plastic through a row of die openings to form filaments and contacting the extruded filaments with high velocity sheets of converging hot air. The converging air contacts and attenuates or draws the filaments down,  
30 depositing them as fibers onto a collector in a random pattern, forming a meltblown web. The meltblown webs have average fiber size between 0.5 to 15 microns which is substantially smaller than the average fiber size of the spunbond web. Another difference between the

meltblown and spunbond webs is that the meltblown webs are generally held together by fiber entanglement with some thermal bonding whereas the spunbond webs are generally thermal bonded by the calender, although  
5 spunbond webs are also bonded by chemical, adhesive, and needling processes.

Both the spunbonding and meltblowing techniques are well known in the art. For example, U.S. Patent 4,405,297 discloses a spunbond process and U.S. Patent  
10 3,978,185 discloses a meltblowing process, both of which are incorporated herein for reference.

For purposes of the present invention, the nonwoven webs can be made of any synthetic thermoplastic polymer used in meltblowing or spunbond processes. By way of  
15 example, these include the following: polyolefins, particularly ethylene and propylene homopolymers and copolymers (including EVA and EMA copolymers), nylon, polyamines, polyester, polystyrene, poly-4-methylpentene, polymethylmethacrylates, polytrifluorochloro-ethylene,  
20 polyurethanes, polycarbonates, silicones, polyphenylene sulfide, and polypropylene or polyethylene terephthalate.

The most common polymers used in spunbonded fabrics include polypropylene having a melt flow rate of 12-40. These polymers generally are extruded at temperatures  
25 ranging from 180° to 350°C.

The most common polymers used in meltblown fabrics include polypropylene, having melt flow rates of 10-2500.

These polymers are generally extruded at  
30 temperatures of between 180 to 350°C and contacted with high velocity air from 180 to 375°C.

The preferred weight of the nonwoven web is from about 0.1 to about 2 oz. per square yard. The preferred

weights are between about 0.25 to about 1.5 oz. per square yard for the spunbond webs.

The nonwovens may include additives to impart desired properties to the webs. Examples of these  
5 additives are wetting agents, fluorochemicals, antistatics, and anti-microbial agents.

As indicated above, the nonwoven webs in a preferred embodiment comprise the outer two layers of the four layer structure. These webs impart strength to the  
10 structure, improve the hand (softness to the feel) and wearability, and improve the comfort and appearance giving the composite fabric a clothlike appearance and feel.

#### Microporous Film

15 The term "microporous film" means microporous membrane. The terms film and membrane are used interchangeably herein.

Microporous films are defined as having a narrow pore sized distribution in the submicron range, from 1.0  
20 to 10 microns. The microporous films can be made by a number of processes, which include (a) dissolving polymers in solution followed by extraction of the solvent by water vapor, (b) stretching of crystallizable polymers which results in micro-sized tears, and (c)  
25 stretching of a mineral filled polyolefin film. The polymers used in the microporous films include PTFE, polyolefins, polyurethanes, polyamides, and polyesters.

The preferred microporous film used in the present invention is a polyolefin prepared by stretching a highly  
30 filled polyolefin film to impart permeability therein, in accordance with U.S. Patent 4,777,073, the disclosure of which is incorporated herein by reference. The microporous film prepared by this process exhibits excellent breathability, at least 3,000, and generally

from 4,000 to 10,000 grams per square meter per day, and in comparison to other microporous film is inexpensive.

- Polyolefins used to make the film include polypropylene, copolymers of propylene, homopolymers, and
- 5 copolymers of ethylene and blends thereof. A preferred polyolefin is a copolymer of polypropylene and low density polyethylene, particularly linear low density polyethylene (LLDPE). The preferred filler at
- 10 concentrations of from 30 to 70 wt% include inorganic fillers such as calcium carbonate, TiO<sub>2</sub>, talc, clay and silica diatomaceous earth, magnesium carbonate, barium carbonate, magnesium sulfate, and the other inorganic fillers listed in the above reference, U.S. Patent 4,777,073.
- 15 Calcium carbonate is the preferred filler. The pore size of this film ranges from 0.1 to 0.5 microns.

#### Staple Fiber Web

- The term "Staple Fibers" as used herein includes natural or synthetic discrete fibers having a length from
- 20 less than 1 inch to about 8 inches, preferably from about 0.5 inch to about 5 inches and most preferably from about 1 inch to about 3 inches. The staple fibers may include only one type of fibers or may include blends. For purposes of the present invention, the fibers must
- 25 exhibit at least some hydrophilic properties. The preferred concentration of the hydrophilic fibers in the blend should be at least 25% and preferably more than 50%.

- The synthetic man-made fibers may be made from
- 30 thermo-plastics such as polyolefins (including polypropylene and polyethylene), polyesters, and polyamides which are extruded to the proper diameter (usually from 10 to 50 microns) and cut in the desired length, usually from 0.5 to 5.0 inches. The staple or

natural fibers may be any cellulosic base fibers, such as cotton, ramie, hemp, flax, jute, kenaf, bagasse, eucalyptus, rayon, and combinations thereof but do not include wood fiber. The staple fibers may be formed into a web by any of the presently known processes, including, but not limited to, thermal bonding, latex bonding, or carding, or needle-punching, or hydroentangling. The preferred web of staple fibers includes cotton fibers or cotton fibers blended with other staple fibers. The cotton fibers preferably have a fineness of between about 3 to 5 micronaire units to give the web flexibility. The cotton staple fibers have an average width of about 15 to 20 microns.

The staple fibers, which exhibit absorbency for aqueous based materials such as water and blood, act as a reservoir for any liquid or blood. The wicking property of the hydro-philic layer also absorbs water vapor. Moreover, the staple fiber web improves the comfort property of the composite.

#### Adhesive

Any of the adhesives compatible with polyolefins and the staple fibers may be used. The preferred adhesives are the hot melt adhesives such as the polypropylene based adhesives, and EVA adhesive, (e.g. 20-40 wt% VA).

#### Adhesive Bonding

It is important in laminating the composites of the present invention to use nonthermal bonding techniques. Thermal bonding has a tendency to damage the microporous film by introducing pinholes. The preferred technique for applying the adhesive is by melt-spraying or meltblowing of adhesives wherein an air/adhesive spray is deposited on one of the surfaces to be bonded. Both the meltblowing or meltspraying involve extruding a filament

or filaments of a hot melt adhesive from a die and contacting the filament or filaments with air to either stretch or attenuate the filament or break it up into droplets which are deposited on the surface of the web.

- 5 The amount of adhesive deposited on the web may vary within a wide range, but it should be sufficient to ensure good adhesion but not so much that substantial amounts of the pores of the web are plugged. Application from about 1 to 10 grams per square meter of the adhesive  
10 should be sufficient, with 1 to 5 grams per square meter being preferred.

As indicated above, the bonding should be by nonthermal pressure techniques. "Nonthermal" means the composite is formed by applying a bonding pressure at  
15 temperatures below melting point or softening temperature of the polymers used in the laminate. With polyolefins, this means that the temperatures are carried out below 100°C and preferably below 50°C, most preferably below 30°C. The lower limit of the laminating temperature will  
20 be ambient which, depending on the geographic location, may vary widely from 0°C to 50°C.

A laminating apparatus 10 which may be used in lami-nating the composite of the present invention is schematically illustrated in Figure 1. The laminating  
25 assembly comprises a plurality of spindles 11, 12, 13, and 14 for receiving rolls of web and film, guide and tension rollers 15, meltblowing dies 16, 17, and 18, calender rolls 19 and 20, and take-up spindle 21. The various rolls may be mounted on the feed spindles 11-14  
30 in the manner described below. A roll 22 of nonwoven web is mounted on spindle 11, with web 23 dispensed therefrom, trained around rollers 15, disposed under the die 16, and fed into the nip of calender rolls 19 and 20.  
A roll 24 of microporous film is mounted on spindle 12

with web 25 dispensed therefrom, disposed under die 17, and fed into the nip of calender rolls 19 and 20. A roll 26 of staple fiber is mounted on spindle 13 and web 27 dispensed therefrom and fed into the nip of calender rolls 19 and 20. Finally, a roll 28 of a nonwoven web is mounted on spindle 14 and web 29 dispensed therefrom. Web 29 is trained around rollers 15, passing under meltblowing die 18, and into the nip of calender rolls 19 and 20.

10 The calender rolls may consist of the following combinations:

- (a) driven rolls 19 and 20 having smooth rubber surfaces; or
- (b) roll 19 with a smooth steel surface and roller 20 having an embossed pattern in the shape of a raised geometric shape such as diamond or square. The embossment (area of the raised portions contacting the web passing through the nip) ranges from 5 to 35%, preferably from 10 to 25% of the total surface of the web in contact therewith.

In operation, the four webs 23, 25, 27, and 29 are fed in overlaying relation into the nip of the rolls 19 and 20 as shown in Figure 1 and driven or pulled therethrough around guide roll 15 and wound onto spindle 21 forming composite roll 34.

30 The calender rolls 19 and 20 are maintained at a temperature well below the softening temperature of the polymers, preferably less than 100°C and the pressure at the nip is maintained between about 50 to 150 psi, preferably 75 to 125 psi, using the smooth rolls and about 150 to 250 pounds per linear inch using the combination smooth roll and embossed roll.



As the webs 23, 25, and 29 pass under meltblowing dies 16, 17, and 18, a spray 30, 31, and 32 of adhesives is deposited on a surface of each web. The adhesive deposited on such webs forms a thin discontinuous layer thereon. Deposition of the adhesive in this manner ensures that adhesive will be at the interface of each of the webs as they are passed through the nip of the calender rolls 19 and 20. The speed at which the webs are processed through the nips may vary within relatively wide ranges, but speeds of 5 to 10 meters per minute are sufficient to ensure good bonding.

As illustrated in Figure 2, the composite 34 comprises a first nonwoven web 23 having outer surface 35 and an inner surface 36 adhesively bonded to surface 37 of microporous film 25. The opposite surface 38 of microporous film 25 is adhesively bonded to surface 39 of staple fiber web 27. Likewise, surface 40 of staple fiber web 27 is adhesively bonded to surface 41 of the second nonwoven web 29. Surface 42 of web 29 when used as protective apparel will be in contact with the wearer's body, and outer surface 35 of the composite 34 will be exposed to the environment. Thus, any blood or toxic liquid of the environment coming into contact with the apparel will first have to pass through the nonwoven web 23 and then through the barrier microporous film 25.

The microporosity and the hydrophobic nature of these layers will act as a barrier for the liquid. Any liquid that does pass these barrier layers will enter the hydrophilic staple fiber layer which will act as a reservoir or wicking layer for containing the blood or the liquid. Finally, the inner nonwoven layer 29 acts as a second barrier, though not nearly as effective as the microporous film 25.

A variation of the lamination apparatus 10 used in the process includes basically the same apparatus without spindle 12 and die 17. In this assembly, one pass is required for three-layer laminates and two passes are required for four-layer laminates.

The breathability of the composite 34 permits air and water vapor to pass sequentially through layers 29, 27, 25, 23, and into the environment, thus providing comfort for the wearer. The staple fiber layer 27 provides for the additional function of acting as a wicking material for any perspiration or liquid that might pass from the wearer outwardly.

The following table presents some of the preferred properties or specifications for each layer.

|                       | Preferred Material               | Weight (oz/yd <sup>2</sup> ) Range | Wt% of Composite (%) |
|-----------------------|----------------------------------|------------------------------------|----------------------|
| Nonwoven Web (23)     | SB or MB                         | 0.25-5.0                           | 10-50                |
| MP Film (25)          | Stretched filled polyolefin film | 0.25-1.5                           | 10-50                |
| Staple Fiber Web (27) | Cotton                           | 0.25-2.0                           | 10-50                |
| Nonwoven Web (29)     | MB                               | 0.25-5.0                           | 10-50                |

The composite 34 preferably has a thickness ranging from 0.2 to 1.5 mils, most preferably 0.3 to 1.0 mils, and an MVTR of at least 400 g/m<sup>2</sup>/24 hrs. and most preferably at least 500 g/m<sup>2</sup>/24 hrs. MVTR of 500-1000 g/m<sup>2</sup>/24 hrs. will be satisfactory for most composites. The composite has a bursting strength of at least 10 psi and preferably of at least 15 psi. The composite may be

made in weight ranging from 2.00 to 6.00 oz/yd<sup>2</sup>, preferably 2.5 to 4.0 oz/yd<sup>2</sup>.

#### Uses of Composites

As amply demonstrated by the test results presented below, the composite of the present invention exhibits a combination of properties making it ideally suited for a number of applications:

- (a) The composite is breathable (see MVTR Test results).
- 10 (b) The composite exhibits good barrier to liquids (see Blood and Viral Resistance Test results).
- (c) The composite has a soft, clothlike hand and appearance.
- (d) The composite with the preferred microporous  
15 film (EXXAIRE<sup>®</sup>) costs less than many composites having other microporous films.

A particularly useful application of the composite of the present invention is in medical protective apparel designed to protect the wearer from contact with external  
20 blood or toxic liquids, or to protect the environment from blood or liquid contamination emanating from the wearer.

Other medical and hygiene uses include feminine hygiene absorbents, baby diapers, adult incontinents,  
25 industrial protective apparel, wound dressing, transdermal patches, and the like. Other uses include sportswear, rain gear, footwear, and the like.

Although the reasons for the improved performance (MVTR and Blood and Virus Resistance Tests) of the  
30 composite of the present invention are not fully understood, it is believed that the combination of the microporous film and the nonwoven layers improve barrier properties and the staple fibers provide a hydrophilic

reservoir or wicking layer for the blood or aqueous liquids.

#### EXPERIMENTS

Experiments were carried out to test the properties of laminates made in accordance with the present invention.

Web Materials: The three layer and four layer laminates were made of the following materials:

10 MB - a polypropylene meltblown nonwoven web having an average fiber diameter of between 3.8 to 4.3 microns;

SB - a polypropylene spunbond web having an average fiber diameter of between 23.0 microns;

15 MP - a polyethylene microporous film marketed by EXXON Chemicals Company as "EXXAIRE";

Cotton - staple fiber cotton which has been carded (C), or thermally bonded (TC), or latex bonded (LC);

20 Adh. - a PP based hot melt adhesive marketed by Finley Adhesive, Wauwatosa, Wisconsin, as H2279.

Laminate Process: Laminate structures were made as follows:

25 a) Three layer composites were made by feeding into the nip of counterrotating calender rolls from three separate webs in overlaid relation. Prior to entering the nip the two outer webs were passed under a meltblowing die which meltblew a hot melt adhesive thereon at about 5.0 g/m<sup>2</sup>. The surface of each outer web thus was bonded to the middle web of the three layer structure and the three layer composite was wound into a roll.

30 (b) The four layer composite was made by preparing the three layer composite as described above

followed by the step of feeding into the nip of calender rolls the three layer composite as described above and a fourth web from a fourth web roll. Prior to entering the nip, the 3-layer composite and the fourth web were each passed under a meltblowing die where an adhesive (H2279) was applied at about 5.0 g/m<sup>2</sup>. The surfaces with the adhesive were fed into the nip to bond with each other. The four layer structure was bonded together and wound on a roll.

Smooth calender rolls (SC) or embossed calender rolls (EC) were used to bond the different layers.

The smooth calender consisted of two rolls provided with smooth rubber covers. The embossed calender consisted of a raised diamond pattern steel roll and a smooth steel roll. The raised area of the diamond pattern represented about 14.7% roll area.

In calendering with the smooth rolls, the calender rolls were operated at a nip temperature of about 20°C, a nip pressure of about 80-90 psi and a speed of about 5-10 m/min. The point bonding with the embossed rolls (Kusters Two Roll) was carried out at a nip temperature of 23°C, a nip pressure of 150 or 200 PLI, and a nip speed of 5-10 m/min.

The smooth calendered composites (having four layers) were prepared by two passes through the calender nip. In the first pass, three layers were laminated with only minimal nip pressure. In the second pass, a fourth web was added and then passed through the calender nip at the conditions described above.

In point bonding, the four layers were combined by the two-pass method described above except the nip

pressure in both passes was minimal. These laminates were then bonded by passing through the nip of the embossed calender at conditions described above.

- 5   Laminate Structures: Duplicate multilayered structures were made; the webs of one being adhesively bonded together by the smooth calender (SC) and the other by the embossed calender (EC) as described above.

SERIES I TESTS:

- 10   The SERIES I TESTS were carried out on composites comprising the following layers:

- (a) a web of MB having a weight of 0.50 (Samples 1, 3, 5, 7) or 0.75 (Samples 2, 4, 6, and 8) oz/yd<sup>2</sup>;
- 15   (b) an MP film having a weight of 0.94 oz/yd<sup>2</sup> except for Samples 3 and 4 which was 0.50 oz/yd<sup>2</sup>;
- (c) a web of cotton (C or TC or LC) having a weight of 0.60 (Samples 7 and 8), 0.65 (Samples 5 and 6), or 0.75 (Samples 1 thru 4);
- 20   (d) a web of MB having a weight of 0.50 or 0.75 [same sample distribution as web (a)].

The laminates were adhesively bonded by meltblowing an adhesive onto a surface of layers (a), (b), and (c).

- 25   The SERIES I TEST composites had the following specifications:

| Sample | Composite   | Weight (oz/yd <sup>2</sup> ) |           | Thickness (mm) |           |
|--------|-------------|------------------------------|-----------|----------------|-----------|
|        |             | Smooth Cal.                  | Emb. Cal. | Smooth Cal.    | Emb. Cal. |
| 1      | MB/MP/C/MB  | 2.92                         | 2.92      | 0.616          | 0.643     |
| 2      | same        | 3.54                         | 3.60      | 0.809          | 0.726     |
| 3      | same        | 2.77                         | 2.95      | 0.633          | 0.589     |
| 4      | same        | 3.39                         | 3.75      | 0.746          | 0.703     |
| 5      | MB/MP/TC/MB | 2.89                         | 2.95      | 0.528          | 0.506     |
| 6      | same        | 3.36                         | 3.51      | 0.678          | 0.688     |
| 7      | MB/MP/LC/MB | 3.04                         | 3.01      | 0.484          | 0.529     |
| 8      | same        | 3.48                         | 3.45      | 0.635          | 0.650     |

SERIES II TESTS:

These tests were carried out on four-layer composites as described for Sample 1 in the SERIES I TEST  
5 Samples except a polypropylene spunbond web having a weight of 0.60 oz/yd was used as layer (a). The SERIES II composites had the following specifications: \_\_\_\_\_



| <u>Sample</u> | <u>Composite</u> | <u>Weight (oz/yd<sup>2</sup>)</u> |                  | <u>Thickness (mm)</u> |                  |
|---------------|------------------|-----------------------------------|------------------|-----------------------|------------------|
|               |                  | <u>Smooth Cal.</u>                | <u>Emb. Cal.</u> | <u>Smooth Cal.</u>    | <u>Emb. Cal.</u> |
| 9             | SB/MP/C/MB       | 3.42                              | 3.30             | 0.695                 | 0.633            |
| 10            | same             | 3.45                              | 3.60             | 0.808                 | 0.740            |

SERIES III TESTS:

These tests were carried out using four layer composites as described in the SERIES I TESTS except layers (a) and (d) were polypropylene spunbond webs having a weight of 0.60 oz/yd<sup>2</sup>. The MP film had a weight of 0.50 for all samples except for Sample 11 (0.94 oz/yd<sup>2</sup>). The cotton layer for the samples had weights of 0.75 (Samples 11 and 12), 0.65 (Sample 13), or 0.60 (Sample 14). SERIES III TEST composites had the following specifications:

| <u>Sample</u> | <u>Composite</u> | <u>Weight (oz/yd<sup>2</sup>)</u> |                  | <u>Thickness (mm)</u> |                  |
|---------------|------------------|-----------------------------------|------------------|-----------------------|------------------|
|               |                  | <u>Smooth Cal.</u>                | <u>Emb. Cal.</u> | <u>Smooth Cal.</u>    | <u>Emb. Cal.</u> |
| 11            | SB/MP/C/SB       | 3.33                              | 3.30             | 0.655                 | 0.870            |
| 12            | same             | 3.13                              | 3.27             | 0.799                 | 0.792            |
| 13            | SB/MP/TC/SB      | 3.24                              | 3.48             | 0.633                 | 0.591            |
| 14            | SB/MP/LC/SB      | 3.16                              | 3.16             | 0.573                 | 0.550            |

COMPARATIVE SAMPLES:

Six laminates without the cotton web and three laminates without the MP film were made and tested. Layers (a), (b), and (d) of Samples 15, 16, 17, 18, 19, and 20 corresponded to the same layers of Samples 1, 3, 2, 4, 9, and 11, respectively. Layers (a), (c), and (d) of Samples 21, 22, and 23 corresponded to the same layers of Samples 1, 2, and 3, respectively. These comparative samples had the following specifications:

| Sample | Composite | Weight (oz/yd <sup>2</sup> ) |           | Thickness (mm) |           |
|--------|-----------|------------------------------|-----------|----------------|-----------|
|        |           | Smooth Cal.                  | Emb. Cal. | Smooth Cal.    | Emb. Cal. |
| 15     | MB/MP/MB  | 2.15                         | 2.21      | 0.369          | 0.453     |
| 16     | same      | 2.12                         | 2.30      | 0.348          | 0.464     |
| 17     | same      | 2.71                         | 2.74      | 0.605          | 0.535     |
| 18     | same      | 2.65                         | 2.60      | 0.512          | 0.482     |
| 19     | SB/MP/MB  | 2.36                         | 2.12      | 0.454          | 0.367     |
| 20     | SB/MP/SB  | 2.48                         | 2.45      | 0.503          | 0.546     |
| 21     | MB/C/MB   | 1.86                         | 1.98      | 0.520          | 0.638     |
| 22     | MB/C/MB   | 2.39                         | 2.39      | 0.692          | 0.674     |
| 23     | MB/C/MB   | 2.12                         | 2.04      | 0.629          | 0.657     |

TEST PROCEDURES:

The following tests were performed on each sample:

Bursting Strength: INDA Standard Test  
(INDA, Association of the  
Nonwoven Fabrics Industry)  
1st 30.0 - 70 (R82)

MVTR  
(Moisture Vapor  
Transmission  
Rate): ASTM E96-80

Resistance of Protective  
Clothing Materials to

Synthetic Blood: (ASTM Designation: ES 21-92):  
This test method covers the  
determination of the resistance  
of protective clothing  
materials to penetration by  
biological liquids using  
synthetic blood under the  
condition of continuous liquid  
contact. Protective clothing  
material "pass/fail"  
determinations are based on  
visual detection of synthetic  
blood penetration.

Resistance of Pro-  
tective Clothing  
Materials to Pene-  
tration by Blood-  
Borne Pathogens  
Using Viral Pene-

tration as a Test  
System:

(ASTM Designation: ES 22-92)

This test method is used to measure the resistance of protective clothing materials to penetration to blood-borne pathogens by using a surrogate microbe under the condition of continuous liquid contact. Protective clothing "pass/fail" determinations are based on detection of viral penetration.

TEST RESULTS: The following Table presents the results  
of the tests carried out:

| SERIES | Composite   | Sample            | Bursting Strength |  | MVTR<br>(g/m <sup>2</sup> /24 hr) | Resistance to Blood<br>(Pass/Fail) |  | Resistance to Virus<br>(Pass/Fail) |  |
|--------|-------------|-------------------|-------------------|--|-----------------------------------|------------------------------------|--|------------------------------------|--|
|        |             |                   | psi               |  |                                   |                                    |  |                                    |  |
| I      | MB/MP/C/MB  | 1 SC <sup>1</sup> | 17.3              |  | 616                               | Pass                               |  | -                                  |  |
|        |             | 1 EC <sup>2</sup> | 16.0              |  | 589                               | Fail                               |  | -                                  |  |
|        |             | 2 SC              | 23.0              |  | 659                               | Pass                               |  | Pass                               |  |
|        |             | 2 EC              | 19.5              |  | 633                               | Pass                               |  | -                                  |  |
|        |             | 3 SC              | 18.3              |  | 718                               | Pass                               |  | Fail                               |  |
|        |             | 3 EC              | 16.0              |  | 726                               | Pass                               |  | Fail                               |  |
|        |             | 4 SC              | 21.5              |  | 716                               | Pass                               |  | -                                  |  |
|        |             | 4 EC              | 18.5              |  | 699                               | Pass                               |  | -                                  |  |
| I      | MB/MP/TC/MB | 5 SC              | 18.3              |  | 690                               | Pass                               |  | Pass                               |  |
|        |             | 5 EC              | 19.0              |  | 610                               | Fail                               |  | -                                  |  |
|        |             | 6 SC              | 28.8              |  | 678                               | Pass                               |  | Pass                               |  |
|        |             | 6 EC              | 23.5              |  | 635                               | Pass                               |  | Fail                               |  |



| SERIES | Composite   | Sample | Bursting        |  | MVTR<br>(g/m <sup>2</sup> /24 hr) | Resistance              |                         |
|--------|-------------|--------|-----------------|--|-----------------------------------|-------------------------|-------------------------|
|        |             |        | Strength<br>psi |  |                                   | to Blood<br>(Pass/Fail) | to Virus<br>(Pass/Fail) |
| I      | MB/MP/LC/MB | 7 SC   | 18.5            |  | 680                               | Pass                    | Fail                    |
|        |             | 7 EC   | 19.0            |  | 623                               | Fail                    | -                       |
|        |             | 8 SC   | 26.5            |  | 587                               | Pass                    | Pass                    |
|        |             | 8 EC   | 24.5            |  | 563                               | Fail                    | -                       |
| II     | SB/MP/C/MB  | 9 SC   | 27.0            |  | 603                               | Pass                    | -                       |
|        |             | 9 EC   | 23.5            |  | 584                               | Fail                    | -                       |
|        |             | 10 SC  | 28.0            |  | 591                               | Pass                    | Pass                    |
|        |             | 10 EC  | 21.5            |  | 578                               | Pass                    | -                       |
| III    | SB/MP/C/SB  | 11 SC  | 31.5            |  | 612                               | Pass                    | Fail                    |
|        |             | 11 EC  | 25.8            |  | 620                               | Fail                    | -                       |
|        |             | 12 SC  | 32.5            |  | 735                               | Pass                    | -                       |
|        |             | 12 EC  | 29.5            |  | 693                               | Fail                    | -                       |
| III    | SB/MP/TC/SB | 13 SC  | 33.0            |  | 675                               | Pass                    | Fail                    |
|        |             | 13 EC  | 30.0            |  | 650                               | Fail                    | -                       |

| SERIES | Composite   | Sample | Bursting |     | MVTR<br>(g/m <sup>2</sup> /24 hr) | Resistance              |                         |
|--------|-------------|--------|----------|-----|-----------------------------------|-------------------------|-------------------------|
|        |             |        | Strength | Psi |                                   | to Blood<br>(Pass/Fail) | to Virus<br>(Pass/Fail) |
| III    | SB/MP/LC/SB | 14 SC  | 36.0     |     | 633                               | Fail                    | -                       |
|        |             | 14 EC  | 33.5     |     | 614                               | Fail                    | -                       |

1 smooth calender

2 embossed calender

- test not performed

Comparison: smooth calender vs. embossed calender  
higher bursting strength  
higher MVTR

improved performance in Resistance to Blood Test  
improved performance in Resistance to Virus Test

The structure used in the SERIES I and II TESTS with smooth calendering (SC) gave the best results in terms of resistance to blood and resistance to virus.

Comparative Tests:

| Composite | Sample             | Bursting        |     | MVTR<br>(g/m <sup>2</sup> /24 hr) | Resistance              |                         | Resistance<br>to Virus<br>(Pass/Fail) |
|-----------|--------------------|-----------------|-----|-----------------------------------|-------------------------|-------------------------|---------------------------------------|
|           |                    | Strength<br>psi | psi |                                   | to Blood<br>(Pass/Fail) | to Virus<br>(Pass/Fail) |                                       |
| MB/NP/MB  | 15 SC <sup>1</sup> | 17.5            | 568 | Pass                              | Pass                    | Pass                    | Pass                                  |
|           | 15 EC <sup>2</sup> | 13.0            | 540 | Pass                              | Pass                    | -                       | -                                     |
|           | 16 SC              | 13.8            | 659 | Pass                              | Pass                    | Fail                    | Fail                                  |
|           | 16 EC              | 11.5            | 544 | Pass                              | Pass                    | -                       | -                                     |
|           | 17 SC              | 15.0            | 563 | Pass                              | Pass                    | -                       | -                                     |
|           | 17 EC              | 14.5            | 534 | Pass                              | Pass                    | -                       | -                                     |
|           | 18 SC              | 15.5            | 621 | Pass                              | Pass                    | -                       | -                                     |
|           | 18 EC              | 13.25           | 589 | Pass                              | Pass                    | -                       | -                                     |
| SB/MP/MB  | 19 SC              | 22.5            | 500 | Pass                              | Pass                    | Fail                    | Fail                                  |
|           | 19 EC              | 22.0            | 589 | Fail                              | Fail                    | -                       | -                                     |
| SB/MP/SB  | 20 SC              | 28.0            | 515 | Pass                              | Pass                    | -                       | -                                     |
|           | 20 EC              | 26.0            | 493 | Fail                              | Fail                    | -                       | -                                     |

| Composite | Sample | Bursting<br>Strength | MVTR<br>(g/m <sup>2</sup> /24 hr) | Resistance<br>to Blood<br>(Pass/Fail) | Resistance<br>to Virus<br>(Pass/Fail) |
|-----------|--------|----------------------|-----------------------------------|---------------------------------------|---------------------------------------|
|           |        | psi                  |                                   |                                       |                                       |
| MB/C/MB   | 21 SC  | 12.5                 | 728                               | Fail                                  | -                                     |
|           | 21 EC  | 10.75                | 724                               | Fail                                  | -                                     |
|           | 22 SC  | 14.0                 | 779                               | Fail                                  | -                                     |
|           | 22 EC  | 11.5                 | 773                               | Fail                                  | -                                     |
|           | 23 SC  | 11.8                 | 766                               | Fail                                  | -                                     |
|           | 23 EC  | 9.8                  | 781                               | Fail                                  | -                                     |

<sup>1</sup> smooth calender

<sup>2</sup> embossed calender

- test not performed

It is significant to note that the comparative composites (Samples 15, 16, 17, 18, 19, and 20) vis-a-vis its corresponding composites of the present invention (Samples 1, 3, 2, 4, 9, 11) with cotton, were (a) consistently lower in bursting strength and (b) consistently lower (except for Sample 19 EC) in MVTR.

All of the Comparative Samples (except Samples 19EC and 20EC) without the cotton passed the Resistance to Blood Test but only one of the three tested passed the more rigorous Resistance to Virus Test.

The data clearly demonstrates the effect of staple fibers (e.g. cotton) on the composites' bursting strength and MVTR and suggests that it plays an important role in the Resistance to Blood and Virus.

It required only the testing of three of the Comparative Samples (21, 22, and 23) to demonstrate the importance of the MP film. None of these samples passed the Resistance to Blood Test, and all had low bursting strengths.

Although the composites of the present invention have been described as comprising three or four layers adhesively bonded together, it is to be emphasized that this represents the preferred structure. Variations include adhesively bonding intermediate layers between two or more of the recited layers.

CLAIMS:

We Claim:

1. A laminate comprising:
  - (a) a first inner layer of a microporous film;
  - (b) a second inner layer overlaying the first inner layer and being composed of staple fibers, at least 25 vol% of which are hydrophilic staple fibers; and
  - (c) first and second outer layers of thermoplastic nonwoven webs positioned on opposite sides of the overlaid inner layers, said nonwoven webs having an average fiber or filament size of 0.5 to 30 microns,  
said layers being bonded together by adhesives placed at the layer interfaces.
2. The laminate of claim 1 wherein the second inner layer comprises at least 25 wt% of cotton fibers.
3. The laminate of claim 2 wherein the second inner layer comprises at least 50 wt% of cotton fibers.
4. The laminate of claim 3 wherein the second inner layer comprises 100% of cotton fibers.
5. The laminate of claim 1 wherein the second nonwoven layer is a meltblown layer having an average fiber size of 0.5 to 15 microns.
6. The laminate of claim 1 wherein both of the outer non-woven layers are composed of meltblown webs having an average fiber diameter of from 0.5 to 15 microns.

7. The laminate of claim 1 wherein the microporous film has an average pore size of 0.1 to 10 microns.
8. The laminate of claim 1 wherein the laminate has a thickness of from 0.2 to 1.5 mils and has the following properties: moisture vapor transmission rate of at least 400 g/m<sup>2</sup> per 24 hrs, and a bursting strength of at least 15 psi.
9. The laminate of claim 1 wherein the laminate is capable of passing the Resistance to Blood Penetration Test of ASTM ES 21-92.
10. The laminate of claim 1 wherein the laminate is capable of passing the Resistance to Virus Test of ASTM ES 22-92.
11. The laminate of claim 1 wherein the microporous film is a particle filled polyolefin stretched to provide permeability therein.
12. The laminate of claim 2 wherein the cotton fibers are selected from the group consisting of carded fibers, latex bonded fibers and thermally bonded fibers.
13. The laminate of claim 1 wherein the microporous film represents at least 25 wt% of the laminate.
14. The laminate of claim 1 wherein the adhesive is hot melt adhesive.

15. The laminate of claim 14 wherein the adhesive bonding the layers together is from 1 to 10 grams/m<sup>2</sup>.
16. The laminate of claim 14 wherein the adhesive is in the form of layers which have been sprayed onto the laminate surfaces and thereafter pressure bonded at a temperature less than 100°C.
17. A laminate suitable for medical uses which comprises:
  - (a) a first nonwoven web selected from the group consisting of thermoplastic spunbond and meltblown webs and having an outer surface and an inner surface;
  - (b) a microporous film having an outer surface facing the first nonwoven web and adhesively bonded thereto, and an inwardly facing surface;
  - (c) a staple fiber web comprising at least 25 wt% of hydrophilic fibers, and having an outer surface adhesively bonded to the inwardly facing surface of the microporous film, and an inwardly facing surface;
  - (d) a second nonwoven web selected from the group consisting of thermoplastic meltblown web of spunbond and meltblown webs, and having an outer surface adhesively bonded to the inwardly facing surface of the staple fiber web.



18. The laminate of claim 17 wherein the microporous film is composed of films selected from the group consisting of polyolefins, polyamides, polyurethanes, and PTFE.
19. The laminate of claim 17 wherein the staple fiber web comprises more than 50 wt% of cotton fibers.
20. The laminate of claim 17 wherein the microporous film is prepared by stretching a polyolefin having inorganic filler dispersed thereon to impart sufficient porosity thereto to provide the film with breathability.
21. A method of making a laminate comprising
  - (a) selecting a layer of a first nonwoven web and a layer of microporous film;
  - (b) melt spraying an adhesive to a surface of either the nonwoven web or the microporous film;
  - (c) overlaying the first nonwoven web and the micro-porous film with the sprayed on adhesive disposed therebetween, the microporous film having an inner surface facing the first nonwoven web and an outer surface;
  - (d) selecting a layer of a second nonwoven web and a staple fiber layer;
  - (e) melt spraying an adhesive to either the surface of the second nonwoven web or the staple fiber layer;

- (f) overlaying the second nonwoven web and the staple fiber layer with the sprayed on adhesive disposed therebetween, said staple fiber layer having an inner surface facing the second nonwoven web and an outer surface;
  - (g) spraying an adhesive on either the outward surface of the microporous film or the second nonwoven web;
  - (h) feeding the four layers in overlaid relation through the nip of pressurized counterrotating calender rolls wherein adhesive is at the interface of each of the layers, whereby the layers are pressure bonded together by the adhesives.
22. The method of claim 21 wherein the staple fibers comprise more than 50% wt% of cotton fibers.
23. The method of claim 21 wherein the first nonwoven web is selected from the group consisting of meltblown and spunbond webs and the second nonwoven is a meltblown web.
24. The method of claim 21 wherein the pressure is from 10 to 200 psi.
25. The method of claim 21 wherein the surface of the nip rolls is made of smooth resilient material.
26. The method of claim 25 wherein the surface of both rolls is smooth.
27. The method of claim 26 wherein one of the calender roll surfaces is embossed.

28. The method of claim 21 wherein the adhesive is a hot melt adhesive.
29. The method of claim 21 wherein the temperature of the rolls is maintained at less than 100°C.
30. A protective apparel for covering a body member to protect the body member from contact with blood or extraneous liquids which comprises
  - (a) a first thermoplastic nonwoven layer having an outer surface being disposed outwardly from the body member, and an inner surface facing the body member;
  - (b) a staple fiber layer having an outer surface facing outwardly from the body member, and an inner surface facing the first nonwoven layer and being adhesively bonded to the outer surface of the first nonwoven layer;
  - (c) a microporous film member having an outer surface facing outwardly from the body member and an inner surface facing the outer surface of the staple fiber layer and being adhesively bonded thereto;
  - (d) a second thermoplastic nonwoven layer having an outer surface facing outwardly from the body member and an inner surface adhesively bonded to the outer surface of the microporous film layer.
31. The apparel of claim 30 wherein the staple fiber layer includes staple cotton fibers and the second nonwoven layer comprises a meltblown web.

32. A laminate comprising a core layer of staple cotton fibers and flanking layers of a microporous film and a nonwoven layer selected from the group consisting of thermoplastic meltblown or spunbond fibers or filaments, said flanking layers being adhesively bonded to opposite surfaces of the core layer.

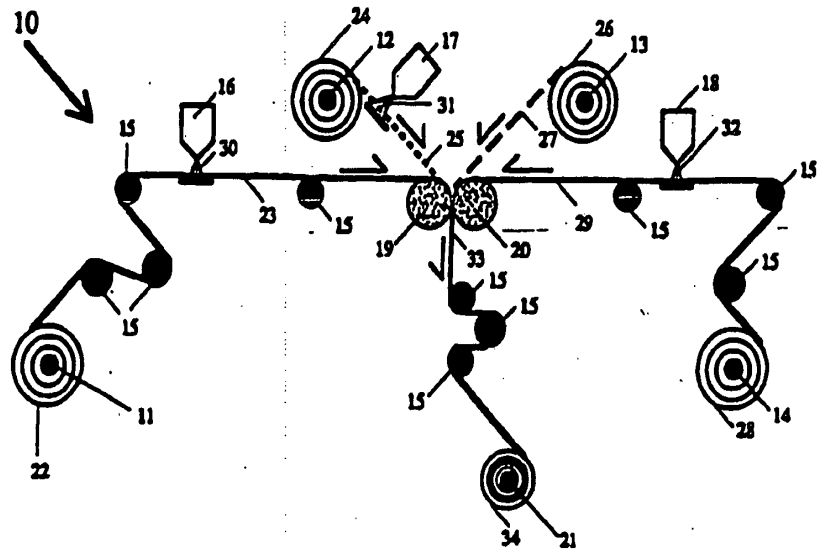


FIGURE 1

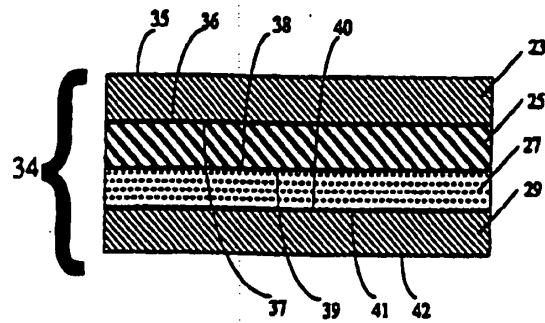


FIGURE 2

# INTERNATIONAL SEARCH REPORT

International Application No.  
PCT/US 95/11865

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 B32B27/12 A61B19/08

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 B32B A61B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category | Citation of document, with indication, where appropriate, of the relevant passages  | Relevant to claim No. |
|----------|---|-----------------------|
| A        | EP,A,0 006 264 (BUCKEY CELLULOSE CORP) 9<br>January 1980<br>see examples 5,6  | 1,17,30,<br>32        |
| A        | EP,A,0 045 137 (SMITH & NEPHEW ASS) 3<br>February 1982<br>see page 2, line 12 - line 19<br>see page 4, line 14 - page 5, line 23<br>see page 9, line 20 - line 23<br>see page 10, line 14 - line 21 | 1,17,21,<br>30        |
| A        | WO,A,93 08024 (FREUDENBERG NONWOVENS LTD.)<br>29 April 1993<br>see claims 1,2,4<br>see page 2, line 30 - page 3, line 15  | 1,2                   |

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents:

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\*O\* document referring to an oral disclosure, use, exhibition or other means

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\*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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Date of the actual completion of the international search

18 December 1995

Date of mailing of the international search report

29.12.95

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# INTERNATIONAL SEARCH REPORT

Int. Appl. No.  
PCT/US 95/11865

| C(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT |  |                       |
|---|--|-----------------------|
| Category *  | Citation of document, with indication, where appropriate, of the relevant passages   | Relevant to claim No. |
| A   | <p>DATABASE VPI<br/>Section Ch, Week 8802<br/>Derwent Publications Ltd., London, GB;<br/>Class A17, AN 88-009323<br/>&amp; JP,A,62 270 639 ( TOYO SODA MFG KK) , 25<br/>November 1987<br/>see abstract</p> | 1,2                   |
| A   | <p>US,A,4 632 860 (D ANTONIO JOHN P ET AL)<br/>30 December 1986</p>  |                       |

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Ind. Int. Application No

PCT/US 95/11865

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